Materials Characterization: SEM/CL & XPS

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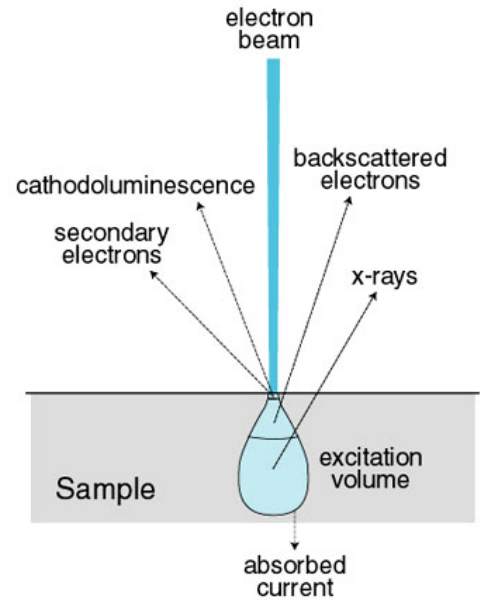
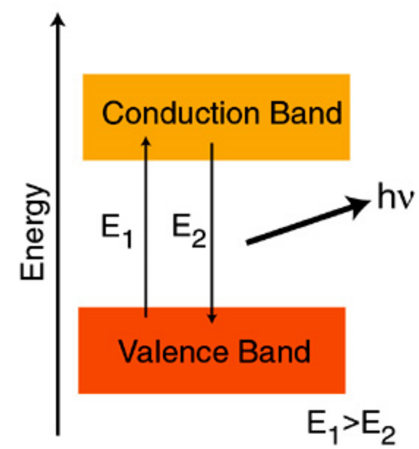
GATOR Engineering
UNIVERSITY OF FLORIDA

SEM & CL

Scanning Electron Microscope (SEM):
•Incident electron beam bombards sample
•Various electron and photon signals are generated
•Amount and type of signal depends on beam energy and excitation volume

Cathodoluminescence (CL):
•Electron-hole pair recombination can result in both phonons and photons
•The photons generated are the basis of CL

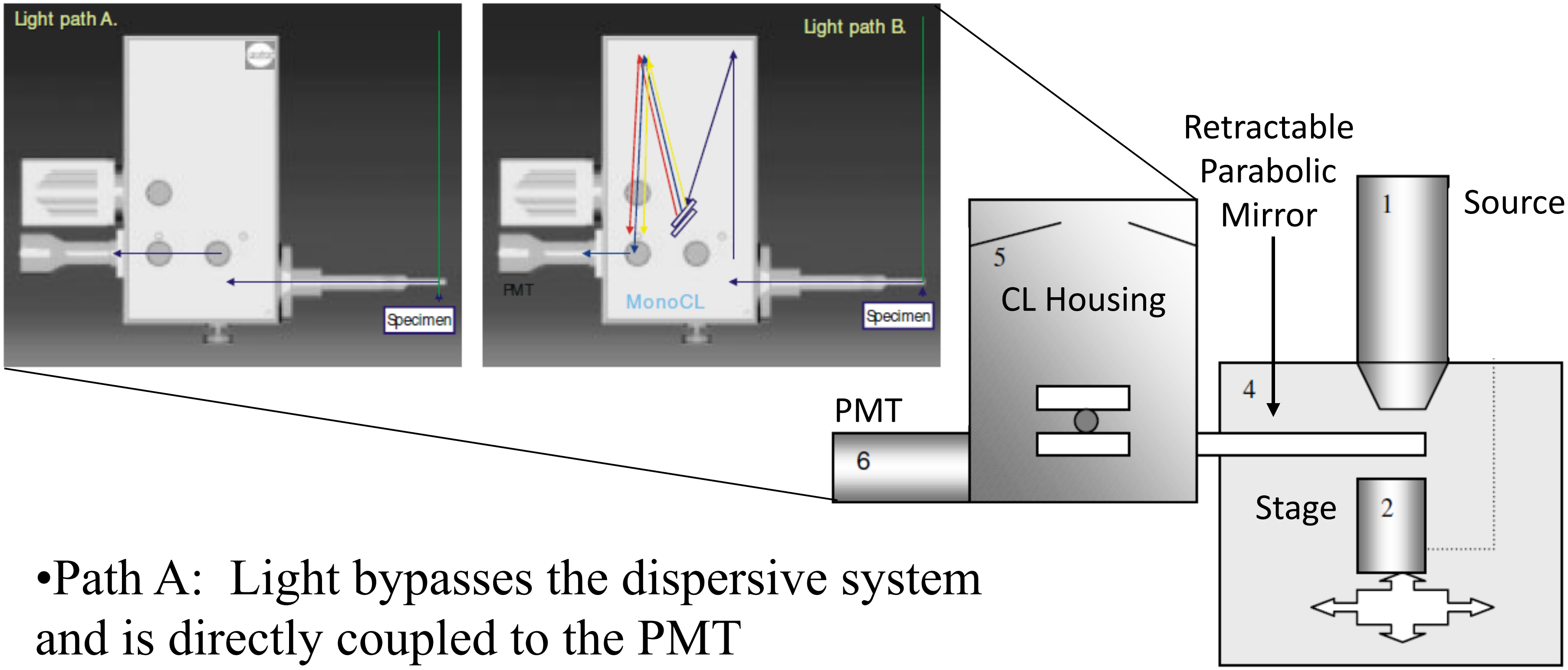




SEM/CL Equipment

FEI NanoSEM 430 with a Gatan MonoCL4 attachment

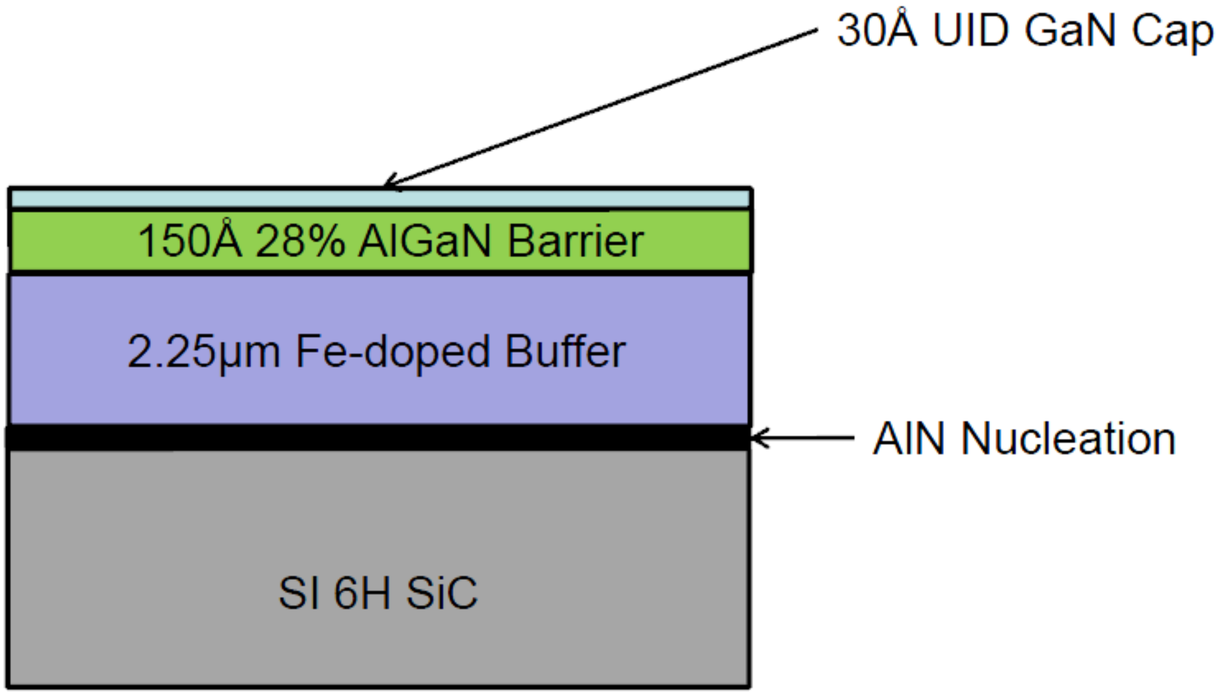
Microscope Set-up & Beam Path



•Path A: Light bypasses the dispersive system and is directly coupled to the PMT

•Path B: Light is coupled through the dispersive system such that a chosen wavelength reaches the PMT

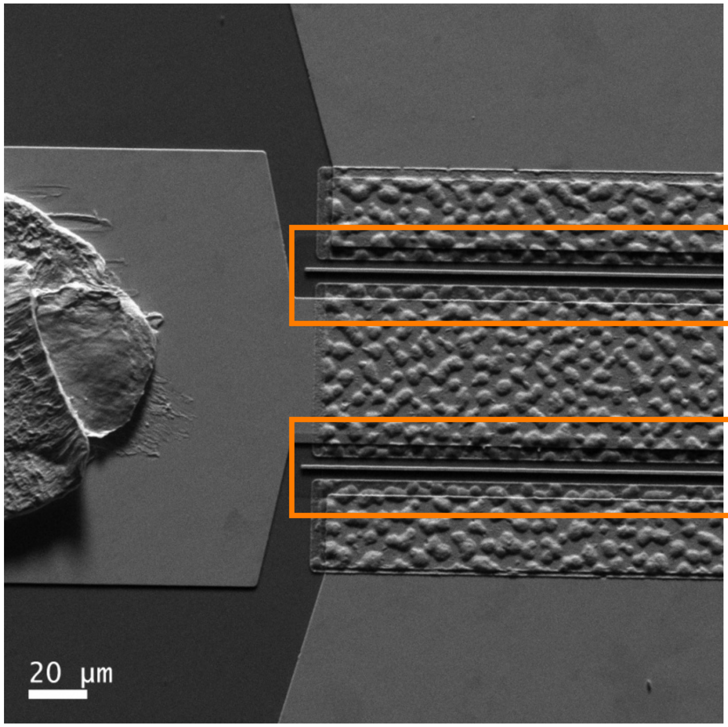
HEMT Device Structure



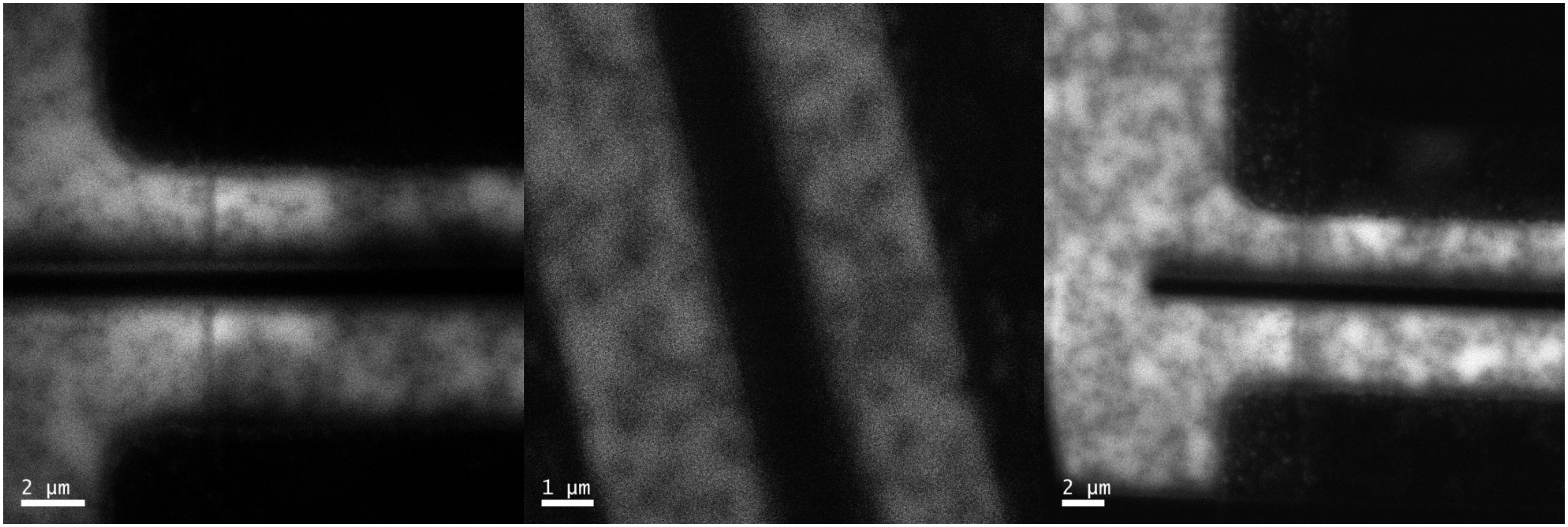
- Unstressed device
- Area analyzed includes T-gate and SiN_x passivation layer

SEM/CL Analysis Conditions

- "Non-destructive" analysis is done before and after failure
- Area of interest is in the channel/ gate region
- Results shown are of depth dependent CL spectra
- Accelerating voltages were varied from 1 kV to 30 kV



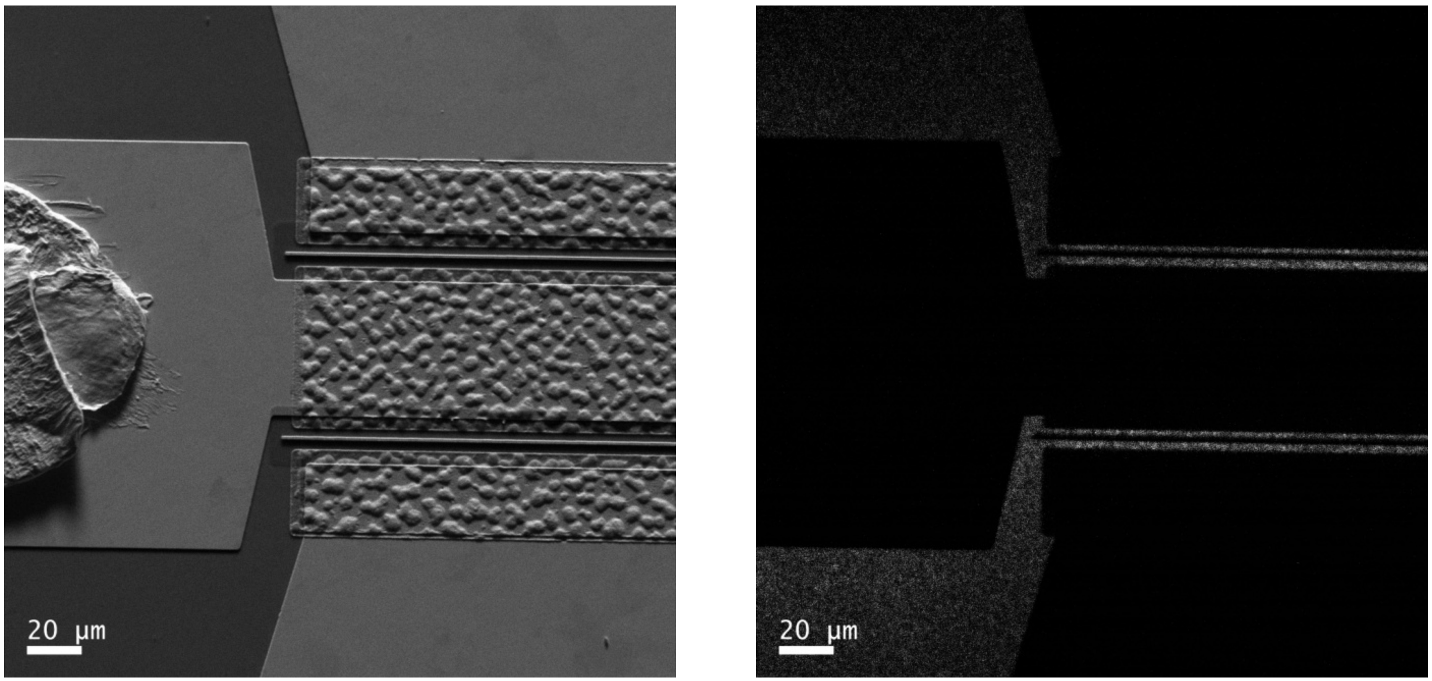
Device Failure CL Image Similarity



- Several devices stressed and failed in different ways all show the same defect structures (i.e. threading dislocations) due to similar contrast
- Accelerating voltages were too high and images are mainly representative of deep buffer layer

SEM/CL Images

Unstressed device was analyzed to form a baseline for microstructural defects for this study



15kV 1000x SE and CL images

Electron Beam Interaction Volume

- The penetration depth of the incident beam varies according to the following equation:

$$R(\mu m) = (0.052 / \rho) * E^{1.75}$$

With: $\rho(GaN) = 6.1 g / cm^3$

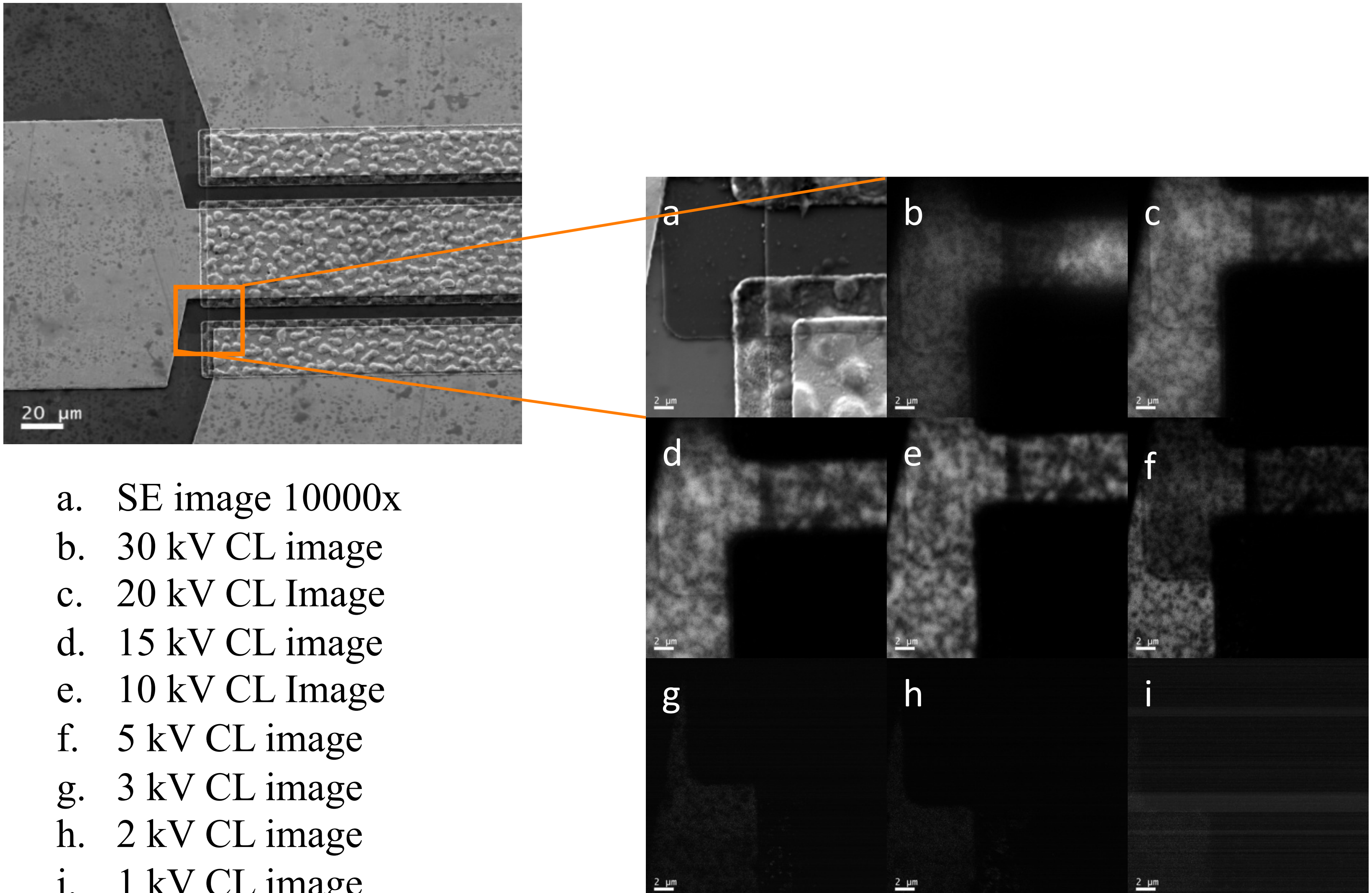
2 kV ~ 30 nm

5 kV ~ 200 nm

30 kV ~ 3200 nm

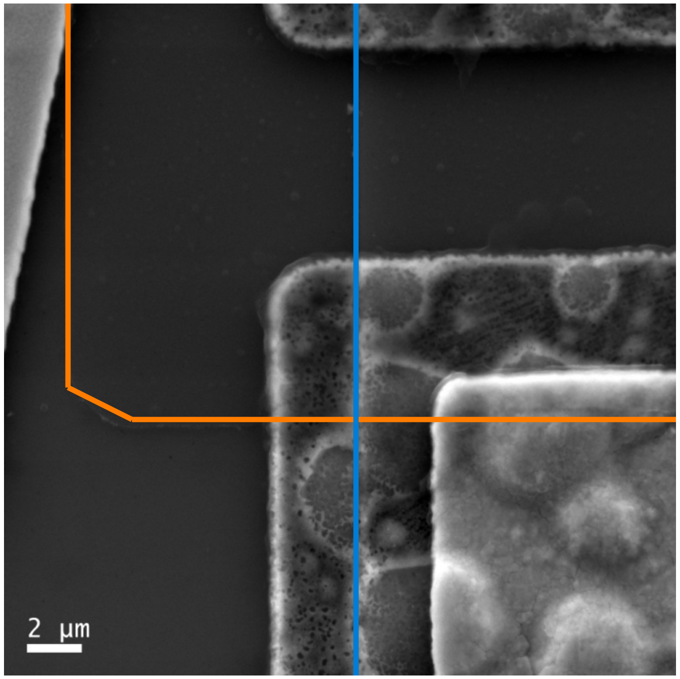
- The thickness of the passivation layer is ~200 nm

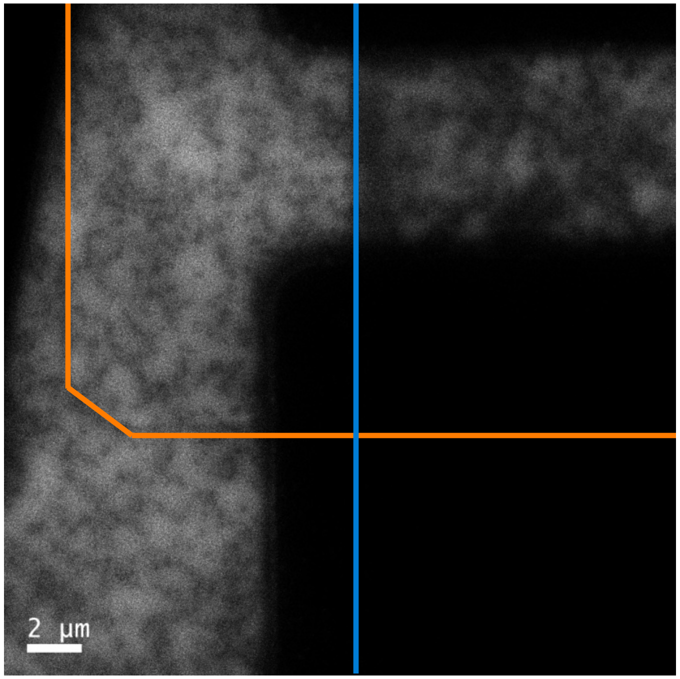
Depth Dependence of CL Signal



a. SE image 10000x
b. 30 kV CL image
c. 20 kV CL Image
d. 15 kV CL image
e. 10 kV CL Image
f. 5 kV CL image
g. 3 kV CL image
h. 2 kV CL image
i. 1 kV CL image

Substrate Defects





20 kV 10000x SE and CL images

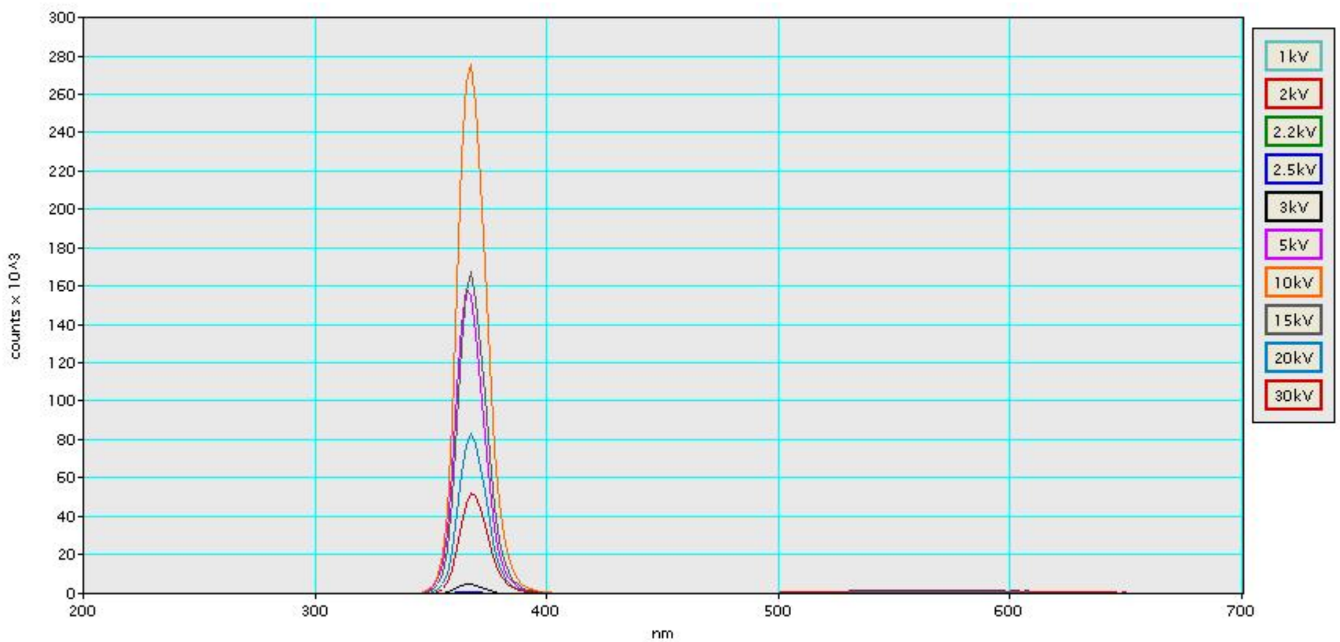
— SiN_x passivation

— Mesa

Monochromatic Spectroscopy

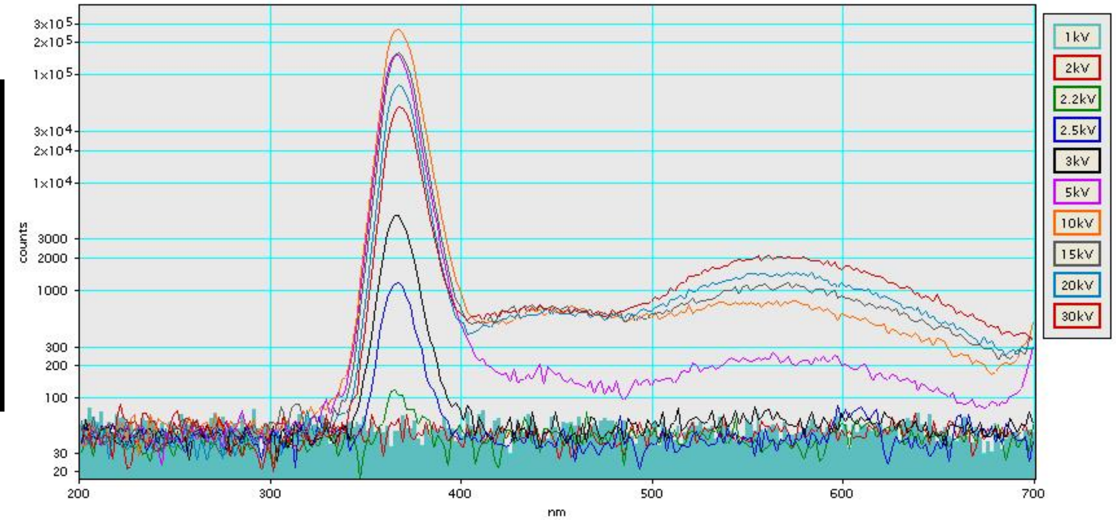
- Accelerating voltages:
  - Varied from 1 kV to 30 kV
  - Penetration depths from 8 nm to >3 mm
- Wavelengths scanned:
  - Spectrum from 200 to 700 nm

GaN band edge shows a maximum signal at 10 kV or ~366 nm



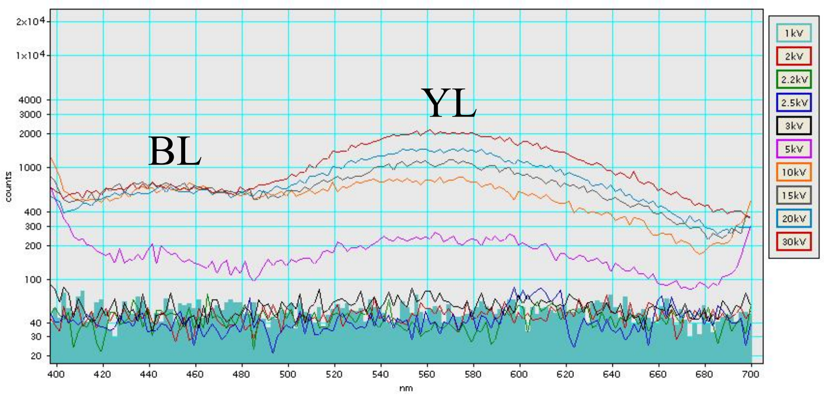
Defect Peaks

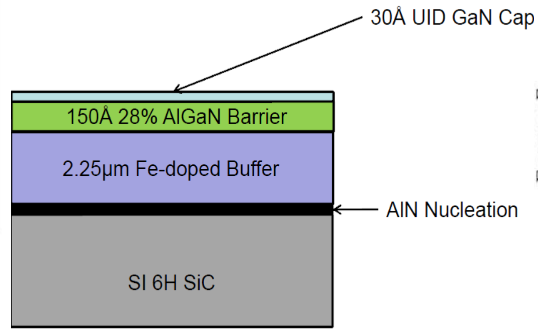
	3 kV	5 kV	10 kV	30 kV
GaN band edge/BL	149.74	749.57	392.83	68.72
GaN band edge/YL	77.14	682.01	374.07	26.87
BL/YL	0.52	0.91	0.95	0.39

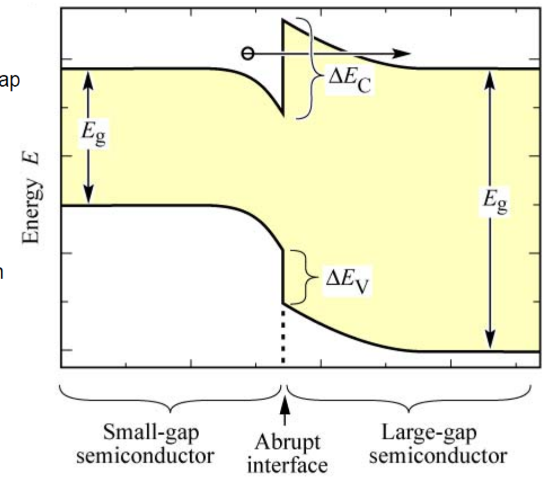


- Visible defect peaks:
  - Blue luminescence (BL) at ~440 nm
  - Yellow luminescence (YL) at ~556 nm
- The BL signal is stationary until we drop to 5 kV while the YL signal increases steadily with voltage

BL & YL Peaks







BL:

•Disappears at 5 kV due to depth profiling within the top AlGaN/GaN layers

•The two most likely causes of the BL signal are:

- •O_{Ga}-related defect
- •Fe from the doped GaN buffer layer

YL:

•Begins to appear at 5 kV and increases with depth (voltage) due to more defects from thicker buffer layer

•Typically associated with V_{Ga}-related defects

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SEM/CL Conclusions & Future Work

Conclusions:

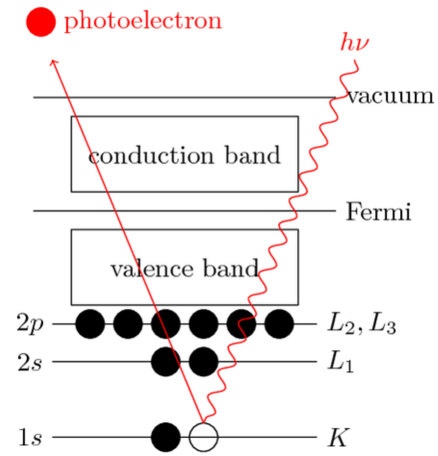
- Threading dislocations are present at all visible accelerating voltages and are unlikely to be sole contributor to YL and/or BL
- BL and YL are depth dependent
- Depth dependence and passivation layer thickness have significant effect on signal strength and wavelengths detected

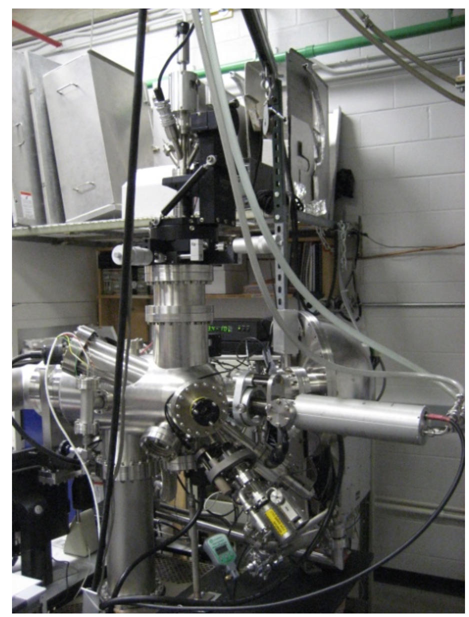
Future Work:

- Sample population of unstressed devices to be stressed will include stripping off passivation layer and gate to view the entire channel clearly through CL to determine cause of failure
- High resolution depth profiling

X-ray Photoelectron Spectrosopy (XPS)

- •AlGaN surface passivation:
    - •Effects of UV ozone exposure time and XPS take-off angle
- •Chemical changes of gate metal contact on GaN under device operation/stressing conditions
    - •300°C anneal in UHV to analyze material behavior under typical device operation conditions
    - •10 Å Ni deposited on GaN (Ga peak still visible)
    - •10 Å Pt deposited on GaN (Ga peak still visible)





Previous Surface Passivation Studies

SiN_x on u-GaN:

- Shift in valence band (VB) of -0.4 eV and Ga 3d peak lost Ga-O portion after vacuum anneal at 200°C
- VB offset recovers to initial value after anneal in air at 200°C and Ga 3d peak recovered Ga-O portion
- Peak positions and relative intensities are nearly identical to pre-vacuum anneal values

SiN_x on HEMT:

- Shift in VBM of -0.9eV after vacuum anneal and no determined Ga-O bonding in the Ga 3d peak
- VB offset recovers to initial value after air anneal, similar to SiNx/u-GaN sample

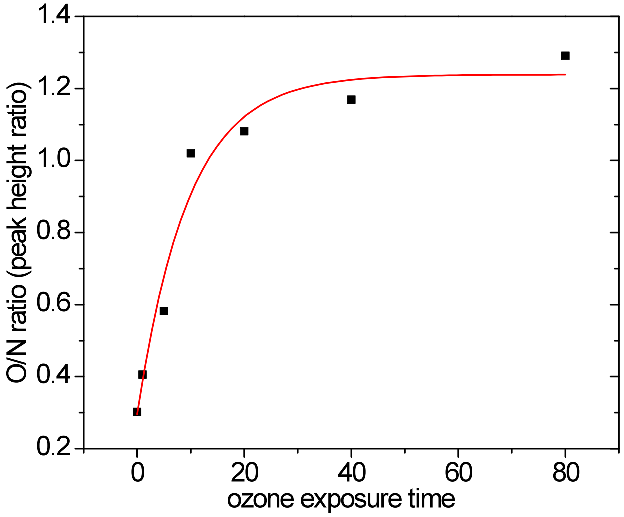
u-GaN:

- Shift in core level to VB only at anneal temp.
- Returns to pre-anneal value upon cool down
- Ga 3d peak shape consistent throughout anneal, no loss of Ga-O portion

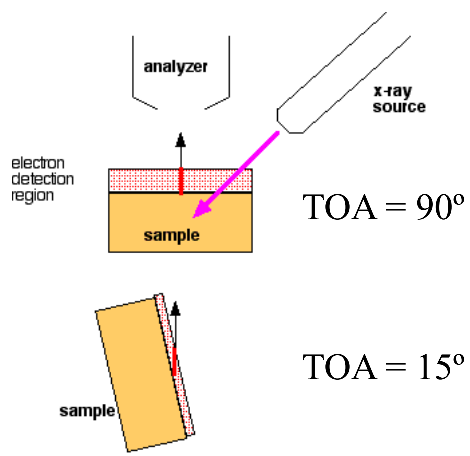
Different mechanism for the different samples due to the significant difference in VB shift.

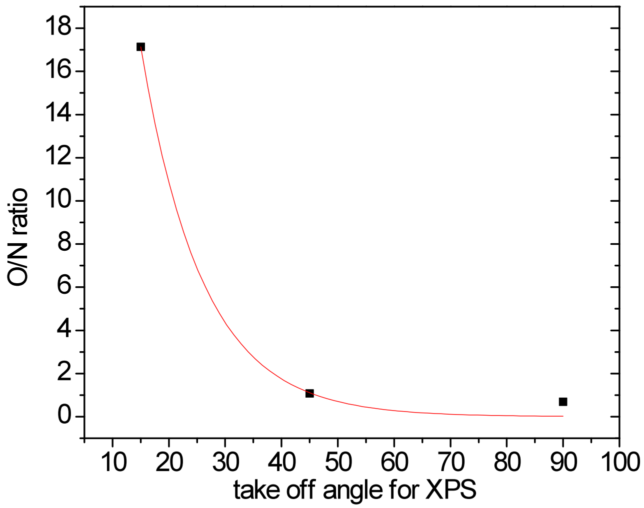
AlGaN Surface Passivation

Ozone effects on AlGaN:
•AlGaN samples placed in UV ozone oven, max. temp. from lamps ~50ºC
•O/N ratio is surface dependent → creation of oxynitride layer
•Future studies of SiN_x passivation layer after ozone exposure



Take-off angle (TOA): angle between energy analyzer and sample surface





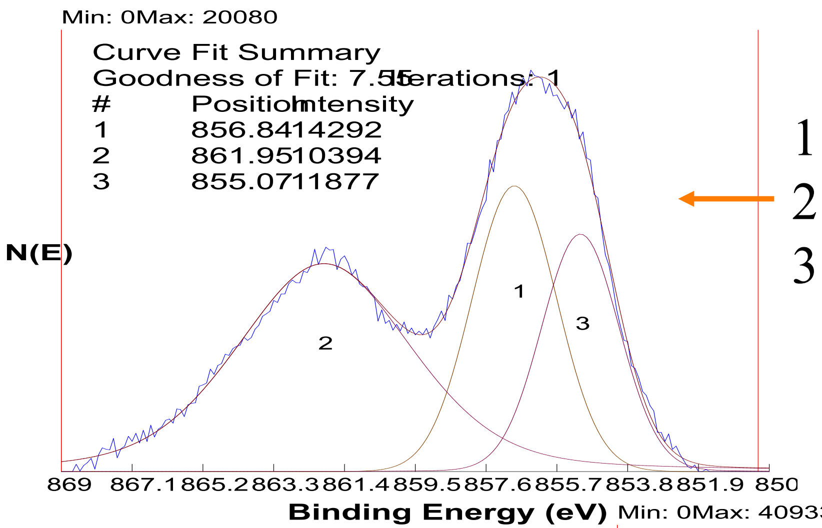
GaN Surface Treatment

Sample Preparation:

- Substrate: 3 µm GaN on sapphire, 2" wafer
- Pretreat:
  - 3 min. 1:1 HCl:DI H_2O, DI H_2O rinse, N_2 dry
  - 25 min. UV ozone exposure
  - 5 min. BOE, DI H_2O rinse, N_2 dry
- MBE anneal: 700°C for 30 min. in UHV
- Wafer cleaved into quarters:
  - One quarter for standard
  - One quarter 10 Å Ni metal deposition
  - One quarter 10 Å Pt metal deposition
  - One quarter set aside for future studies

Ni on GaN Anneal Study

Pre-vacuum anneal

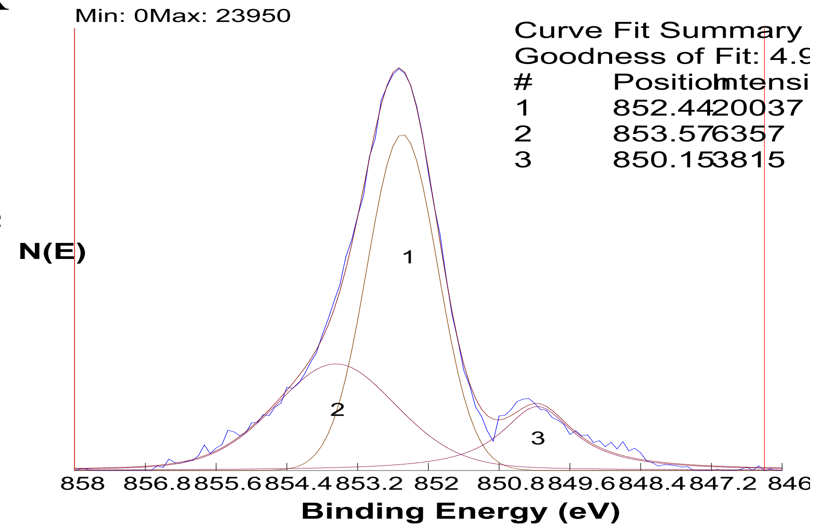


Ni $2p_{3/2}$ Peak

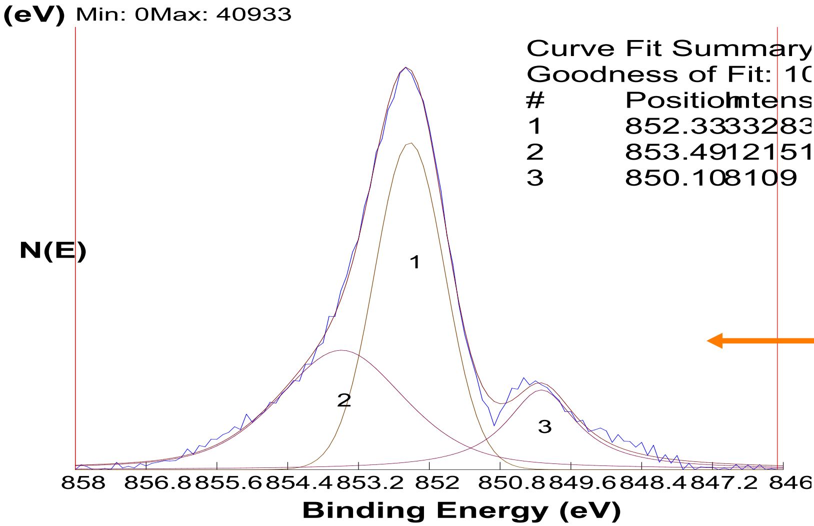
Curves:

1. Ni-O
2. Ni satellite
3. Ni metal

Post-vacuum anneal



Vacuum anneal 300ºC

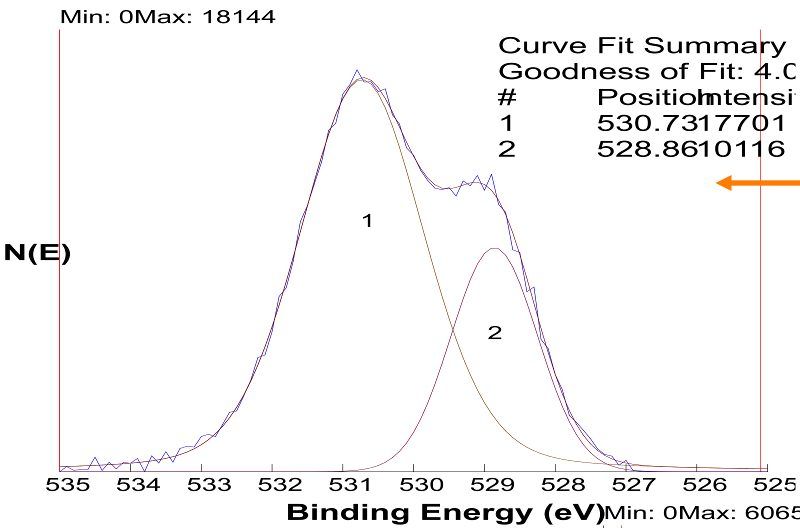


Curves:

1. Ni-O
2. Ni-O satellite
3. Ni metal

Ni on GaN Anneal Study

Pre-vacuum anneal

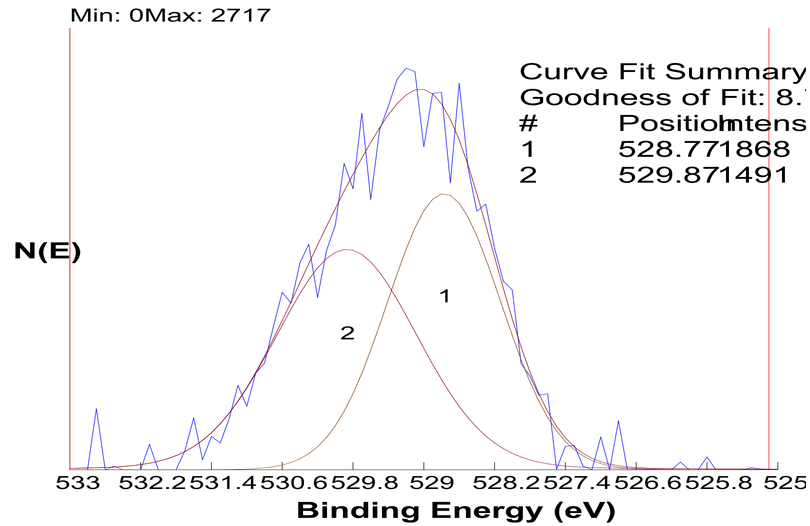


O 1s Peak

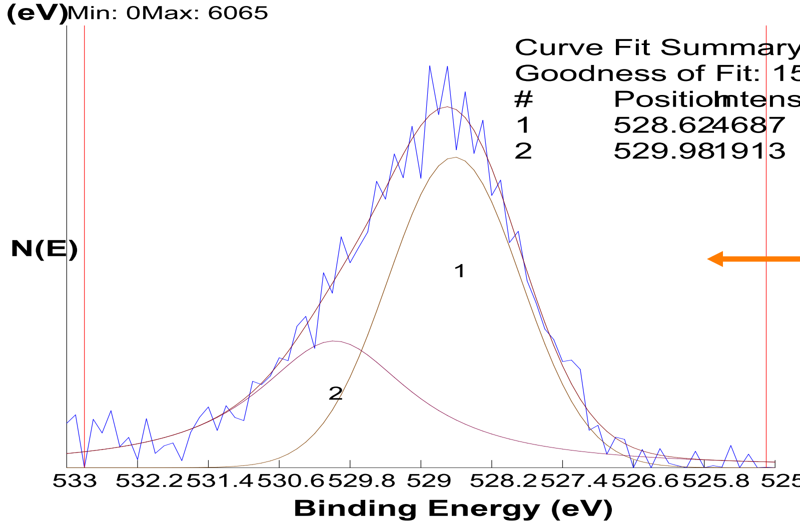
Curves:

1. Ga-O
2. Ni-O

Post-vacuum anneal



Vacuum anneal 300ºC

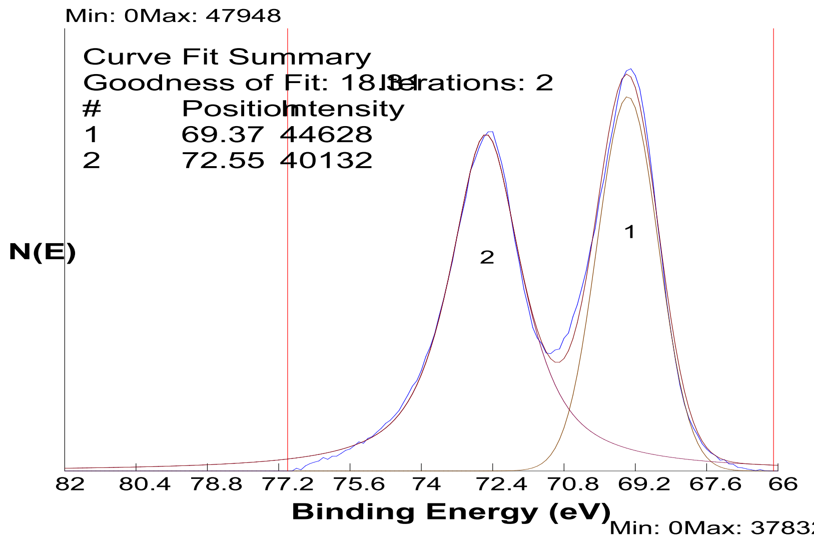


Curves:

1. Ni-O
2. Ga-O

Pt on GaN Anneal Study

Pre-vacuum anneal

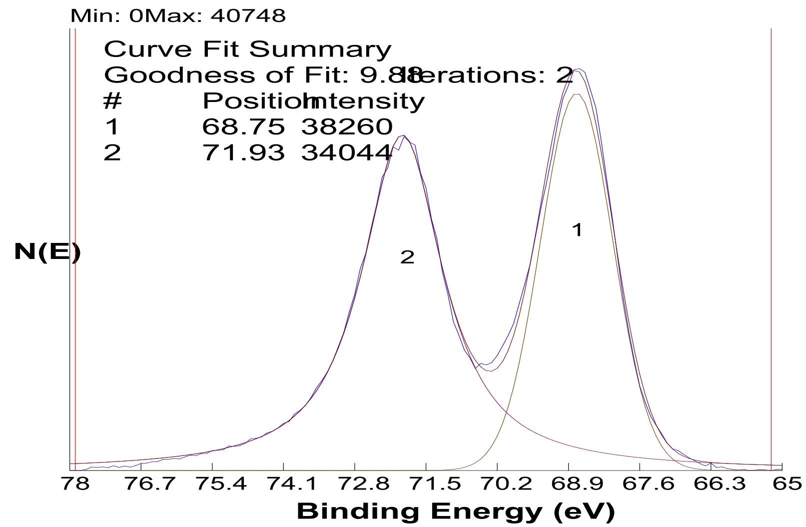


Pt 4f Peaks

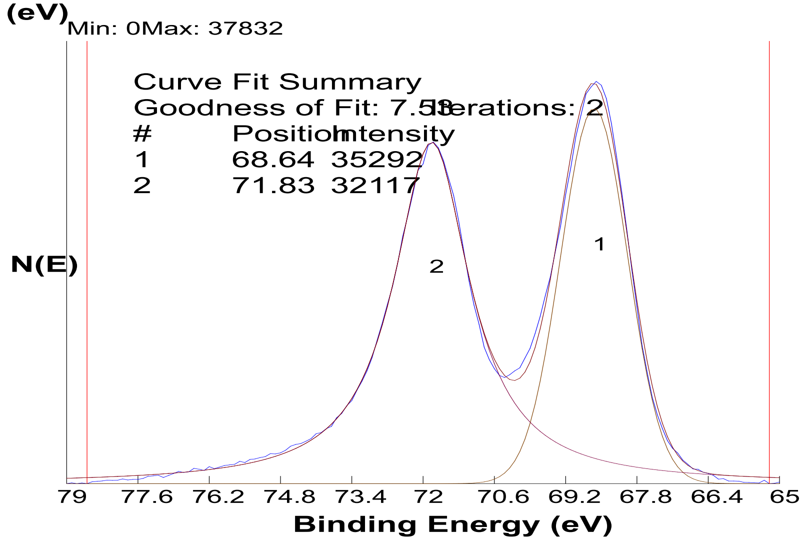
Curves:

1. Pt $4f_{7/2}$
2. Pt $4f_{5/2}$

Post-vacuum anneal



Vacuum anneal 300ºC



XPS Conclusions & Future Work

Conclusions:

•Oxynitride layer on AlGaN possible barrier for SiN_x/AlGaN reaction

•Gate metal/GaN chemical changes due to 300°C anneal in UHV:

- Ni reacts with O from native oxide on GaN → material properties of NiO detrimental to Ni gate contact operation
- Pt does not readily react with O from native oxide on GaN→ better suited for gate contact operation

Future Work:

•AlGaN UV ozone exposure followed by SiN_x passivation → chemical changes due to vacuum anneal

•Introduction of oxynitride layer on GaN before gate metal deposition → chemical changes due to vacuum anneal